

DEGREE OF OPENING-UP OF THE LEATHER STRUCTURE CHARACTERIZED BY ACOUSTIC EMISSION†

by

CHENG-KUNG LIU*, NICHOLAS P. LATONA, AND GARY L. DiMAIO
*United States Department of Agriculture, ** Agricultural Research Service,
Eastern Regional Research Center
600 EAST MERMAID LANE,
WYNDMOOR, PA 19038-8598*

ABSTRACT

Liming is one of the key steps in the leather-making process - to remove hair and non-collagenous materials from the hide and, more importantly, to open up the fibrous structure for tanning and lubrication. However, the leather industry lacks an effective means to identify proper liming conditions that produce a sufficient degree of opening-up. As part of our effort to investigate the applications of acoustic emission technology to leather manufacture, we have examined the feasibility of using the acoustic emission (AE) technique to measure the degree of opening-up of leather produced with various liming times. Leather samples were stretched and contacted with an acoustic sensor to collect various acoustic quantities. Observations showed that a change in the degree of opening-up of the fiber structure associated with the increase of liming time can be detected by a history plot of acoustic emission counts. The samples with less than 24 hours liming show a relatively smooth history plot, whereas the samples with a liming time greater than 24 hours produce a pronounced saw-shaped pattern, and the longer the liming time, the more erratic the pattern becomes. This behavior mirrors the change in the extent of opening-up of the fibrous structure engendered by the lime action. We have concluded that a qualitative association exists between the degree of opening-up of leather and the acoustic counts patterns measured by an AE analyzer. The results of this work may provide a route to monitor the degree of opening-up of leather.

INTRODUCTION

lime (CaO-H₂O) has been used for centuries in the leather-

making processes to swell and to loosen the hide structure, to remove the non-collagenous materials from raw hide and, more importantly, to open up the fibrous structure. The liming process must be done properly to ensure that the hide fiber structure is adequately dilated in order to make it ready for the subsequent processes such as pickling, tanning, and fatliquoring. The current prevailing method to examine the degree of opening-up is by microscopic observation of the extent of fiber separation. The procedures of microscopic examination can be very time consuming and the results are often subjective. We therefore have investigated another route to examine the degree of opening up. It was previously shown by Kronick¹ that there is a close relationship between fiber adhesion and acoustic emission properties. On the other hand, a higher degree of opening up may mean a lesser extent of fiber adhesion, i.e., more fiber separation. Therefore, we have recently investigated the feasibility of using acoustic emission technology to measure the degree of opening-up associated with the liming process.

In our research center, acoustic emission (AE) has been recognized for some years now as a useful method for characterizing leather properties. Kronick and Thayer have demonstrated that the strength of fiber adhesions can be determined by "listening" to the sounds emitted by the sample when it is stretched.¹ Moreover, Kronick and Maleeff reported that by observing a sudden increase in energy and frequency of acoustic pulses in a tensile test, they were able to determine when the leather was about to fail long before it fractured.² The implication of this finding is that we may be able to predict leather strength by AE measurements without damaging the leather, in a so-called nondestructive test. At the USDA laboratory, we then examined the relationship between tensile strength and any of the AE data - number of hits, acoustic energy generated during stress-strain tests, or amplitude distribution. Observations showed an excellent correlation between the tensile strength of

Technical Paper based on a presentation at the 97th

Corresponding Author, e-mail address: cliu@arserrc.gov.

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ather and the corresponding acoustic cumulative energy at break, read from an acoustic-emission analyzer. Moreover, a linear relationship was discovered between the acoustic cumulative energy at break and the initial acoustic cumulative energy when leather was elongated ten percent of its original length. More importantly, a correlation was observed between the initial acoustic cumulative energy and the tensile strength of leather.³ The implication of these phenomena is that the tensile strength of leather may be predicted without breaking the leather by measuring the initial cumulative acoustic energy. The long range goal of this ongoing research project is the production of an AE tester, providing the leather industry with a nondestructive way in which to monitor the quality of leather at each intermediate leather-making stage. As a result, tanners would be able to adjust their leather-making processes accordingly to yield high quality leather.

Besides tensile strength, one of the other important mechanical properties required for leather products, particularly those used for upholstery, is the ability to withstand tearing. Recently, a joint effort of the USDA and BLC was undertaken to use the acoustic-emission method to gain insight into the reason for tear failure. In a tear test, chrome-tanned leather samples were contacted with an acoustic sensor to collect various acoustic quantities. Data showed that the samples stronger in tear strength gave a significantly lower acoustic count. In contrast, the samples with poor tear strength generated much more frequent sound waves, i.e., more acoustic counts.⁴ This seems to be contrary to results from tensile failure tests, where the higher strength leather always produces more total acoustic counts. Observations also showed that harsh drying conditions or thin corium can lead to a brittle structure, which consequently yields poor tear resistance. In an acoustic-emission test, this can be reflected in high acoustic counts because of frequent fiber breaking and friction associated with the brittle structure. All the AE data reported are above the sonic range (>50 Hz), thereby avoiding the problems of room noise. Leather is a natural fibrous material, and any significant fiber movements or fiber breakages will emit sound waves. From analysis of those counts, frequency and energy associated with emitted sound waves during the tensile tests of leather made with various liming time, one may gain a correlation between acoustic emission quantities and the degree of opening up.

EXPERIMENTAL

Materials

Nature bovine hides were used in our liming experiments.

with the total liming time varied from 3.5 hours to 96 hours. Three fresh full hides were cut into six sides, and each side was assigned a different liming time. Three percent lime, two percent sodium sulfide, and one-hundred percent float were used to dehair and lime the sides. In one drum the sulfide was left in with the lime for those sides limed for 24 hours or less (3.5 and 7 hours.) After 24 hours, the remaining sides in another drum were washed and put into 3% lime and 100% float (bath ratio) for the duration of their designated liming times (48, 72, or 96 hours). Next, the hides were delimed, pickled, and chrome-tanned. The leather was then split to a thickness of approximately 2mm. The leather was not retanned to avoid the complication of retannage on the acoustic emission quantities. Then the leather was treated with an anionic fatliquor composed of 10% Coripol DXA-G from Together for Leather (Germany) according to the standard fatliquoring process. Finally the leather was toggle-dried and then allowed to equilibrate in a conditioning room at 23°C and 50% RH. The final moisture content of the leather right before physical testing was 17 ± 1 percent (dry weight basis), determined by a leather moisture meter (Delmhorst Instrument Co.).

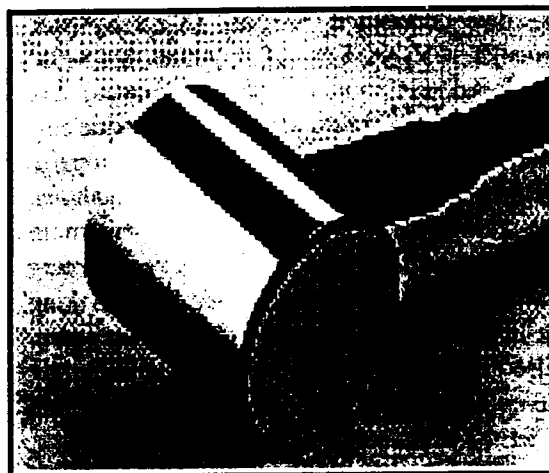
Overview of the AE Theory and Apparatus

The deformation of leather (as leather is squeezed, torn or stretched) caused by an external force is accompanied by a rapid movement, relocation, or breaking of structural elements such as fibrils, fibers and fiber bundles. As a result, sound waves are produced that can be detected by an acoustic transducer and converted into electronic signals. The basic phenomenon may be defined as an acoustic emission event, which is translated by an AE analyzer as a "hit."⁵ A single acoustic emission event (hit) may consist of several emission counts, which are the number of times a signal from the transducer crosses a preset threshold of amplitude, as illustrated in Figure 1d.

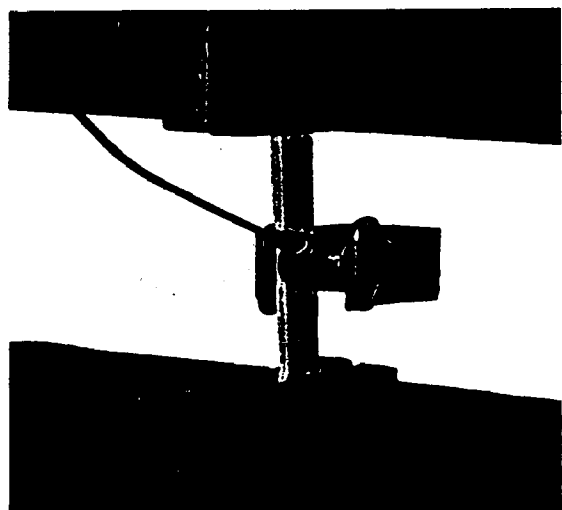
In the tensile test, the leather sample is gripped between two jaws as shown in Figures 1a and 1b. The sample is subjected to a tensile force and slowly stretched at a constant strain rate. Due to this tensile stretching, the leather sample emits sound waves, which are detected by an acoustic transducer. Tensile tests and AE data collections were performed simultaneously to study their relationships directly. To obtain acoustic emission data, a small piezoelectric transducer shown in Figure 1c, resonating at 150 kHz (Model R15, Physical Acoustics Corp., Princeton, NJ), 10mm in diameter and weighing 20g, coated with a film of petroleum grease for more efficient acoustic coupling, was clipped against the leather sample (Figure 1b). Electrical signals emanating from this transducer were processed with a



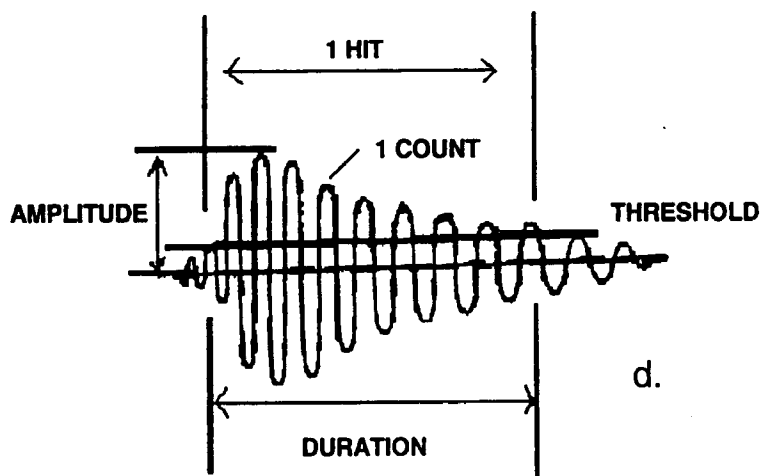
a.



c.



b.



d.

FIGURE 1. - Acoustic emission instrumentation.

Model 1220A preamplifier and a LOCAN-AT Model 3140 acoustic emission analyzer with the LOCAN-AT Upgrade software (Physical Acoustic Corp.). Each acoustic hit (pulse) from an event in the sample caused a damped oscillation to be emitted by the transducer as illustrated in Figure 1d. The wave signal in Figure 1d can be represented by Equation 1, ignoring the initial signal build-up.⁶

$$A(t) = A_0 \exp(-Bt) \sin \omega t \quad (1)$$

Where:

A = Amplitude as a function of time;
 A_0 = initial signal amplitude ;
 B = damping coefficient (greater than 0),
 t = time, and
 ω = angular frequency

The analyzer recorded the arrival time of each oscillation hit, its amplitude, and its energy. Only hits giving maxi-

mum amplitudes greater than 35 dB from the transducer were counted. Each acoustic hit generates a wavetrain from the transducer consisting of a number of oscillations (waves), so-called "acoustic counts." The hits with high amplitudes always produce high numbers of counts. The energy of the hits, estimated by the "ring-down" method, is proportional to the average area under the rectified hits, so energy is determined by the hit amplitudes and the hit durations.⁷

Measurements

The samples were stored in a conditioned room at 23°C and 50 % RH before testing according to ASTM standard method D1610-96. Tensile tests and AE data collections were performed simultaneously on dumbbell-shaped leather samples cut from the standard test area (ASTM D2813-97) with the long dimension parallel to the backbone. Each test was conducted on five samples to obtain an average value.

An Instron mechanical property tester, model 1122, and Testworks 3.1 data acquisition software (MTS Systems Corp., Minneapolis, MN) were used throughout this work. The crosshead speed was reduced from the standard 254 mm/min (10 in/min) to 50 mm/min in order to provide sufficient time for AE data collection. Mechanical property measurements included tensile strength, Young's modulus, and toughness. Tensile strength is defined as the maximum stress. Young's modulus is a physical quantity representing the stiffness of a material. It is determined by measuring the slope of a line tangent to the initial stress-strain curve. Toughness (also called fracture energy) was determined by measuring the energy to fracture the leather sample, which is the area under the stress-strain curve.⁸

Scanning electron microscopic examinations were conducted on the cross section of leather samples to examine the degree of opening up in the fibrous structure. Samples were mounted on aluminum specimen stubs using colloidal silver adhesive (Electron Microscopy Sciences, Ft. Washington, PA) and sputter-coated with a thin layer of gold. Images were collected using a Model JSM 840A scanning electron microscope (JEOL USA, Peabody, MA), integrated with a model Imix 1 digital image workstation (Princeton Gamma-Tech, Princeton, NJ), and operated in the secondary-electron imaging mode.

The shrinkage temperature was determined using a pressure shrink vessel developed by Fein et al.⁹ The samples were

cut to 5.6 mm x 57.2 mm with the long axis parallel to the backbone. The shrinkage temperature was found by reading the temperature at which noticeable shrinkage occurs while gradually heating the leather in distilled water. The reported value is the average of three measurements.

RESULTS AND DISCUSSION

Acoustic Hits vs Time

A basic way to graph AE activities is to plot the rate of hits as a function of time. Figure 2 displays the chronological course of the test, demonstrating the typical hits rate vs time profiles during the tensile testing of leather for two significantly different liming times. Figure 2a shows that the 3.5-hour liming samples emit very little sound for the first-ten second stretch, followed by a steep increase in the hits rate to a peak point at around 25 seconds, then a sudden decrease thereafter. This peak signifies a major fracture of the leather sample. The last peak shown on the figure is probably due to a few fiber bundles still linked together after the majority of the sample fractures as shown in the first peak. In contrast, Figure 2b, of a sample exposed to a longer liming time of 48 hours, shows a steady increase in the hits rate from the beginning of the tensile test to a peak point at about 42 seconds as the leather is fractured. Another significant difference is that the profile of the 48-hour liming sample has a more pronounced saw-shaped pattern and has a slower increase in hit rate as well as a longer fracture time than that

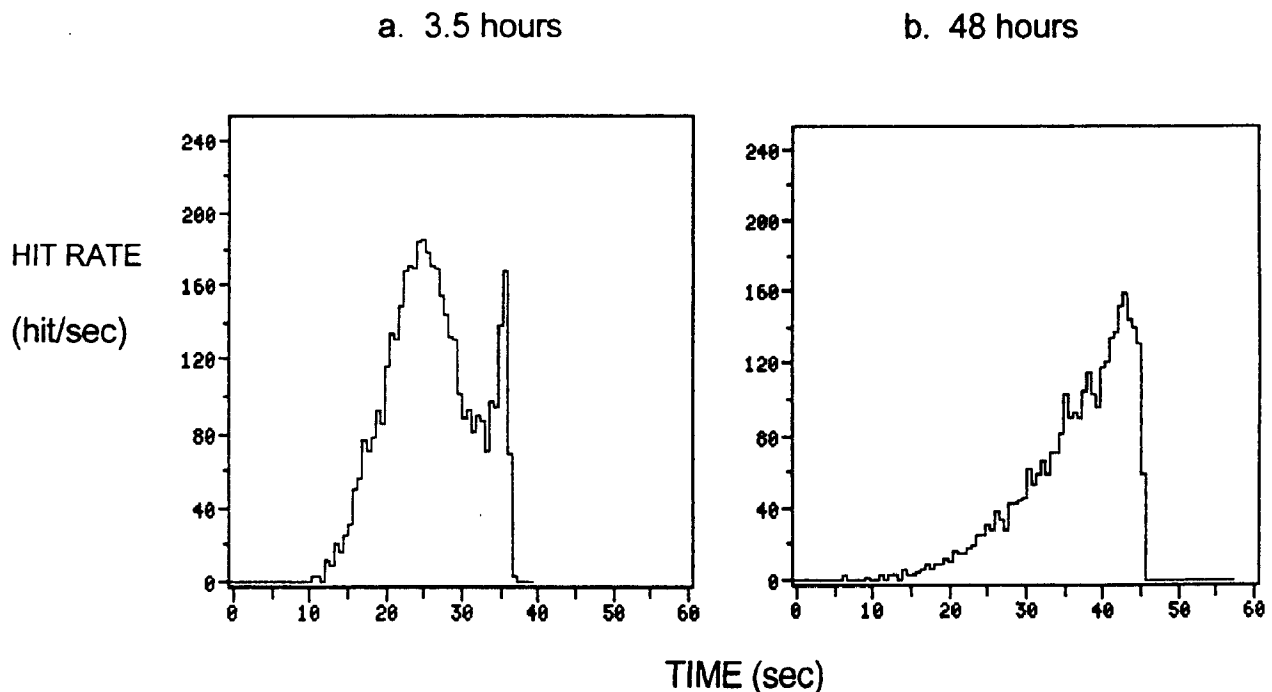


FIGURE 2. - Acoustic hits history.

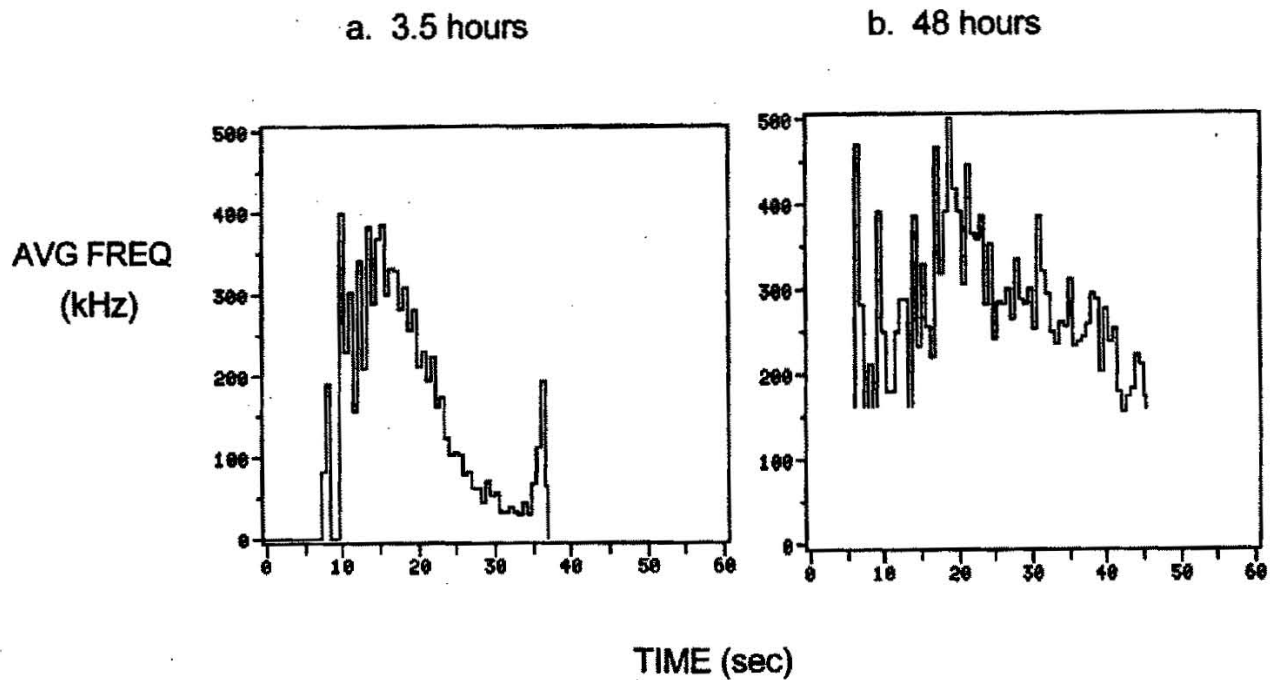


FIGURE 3. - History plot of frequency.

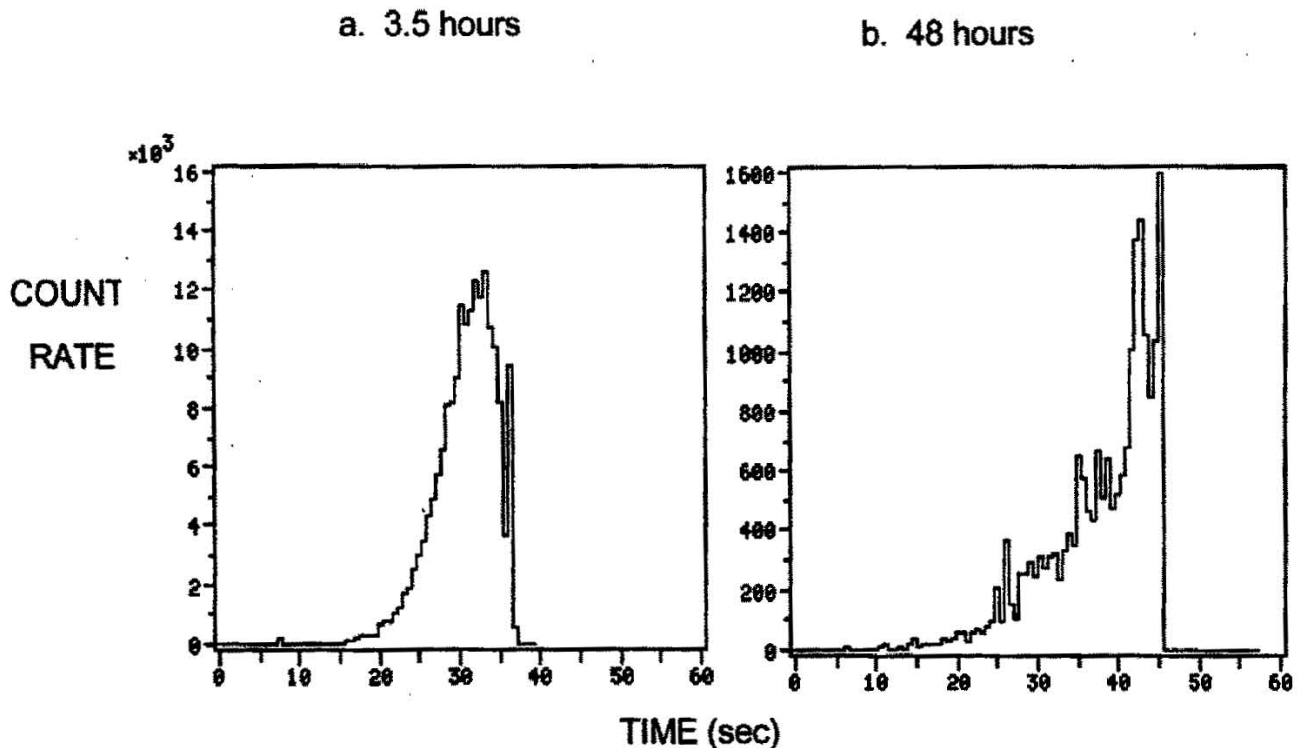


FIGURE 4. - Acoustic counts history.

of the 3.5-hour liming sample. This may be attributed to a better opened-up fiber structure, in that the fibers are more free to move and to aggregate together, engendering a higher stress and emitting more sound. As the leather sample is stretched farther, the aggregated fiber bundles may separate.

This causes the separated fibers to realign and redistribute the total stress among themselves, resulting in a stress relaxation and consequently the emission of less sound. This fluctuation of stress makes the hit-rate curve discrete. The above argument may explain why the 48-hour sample

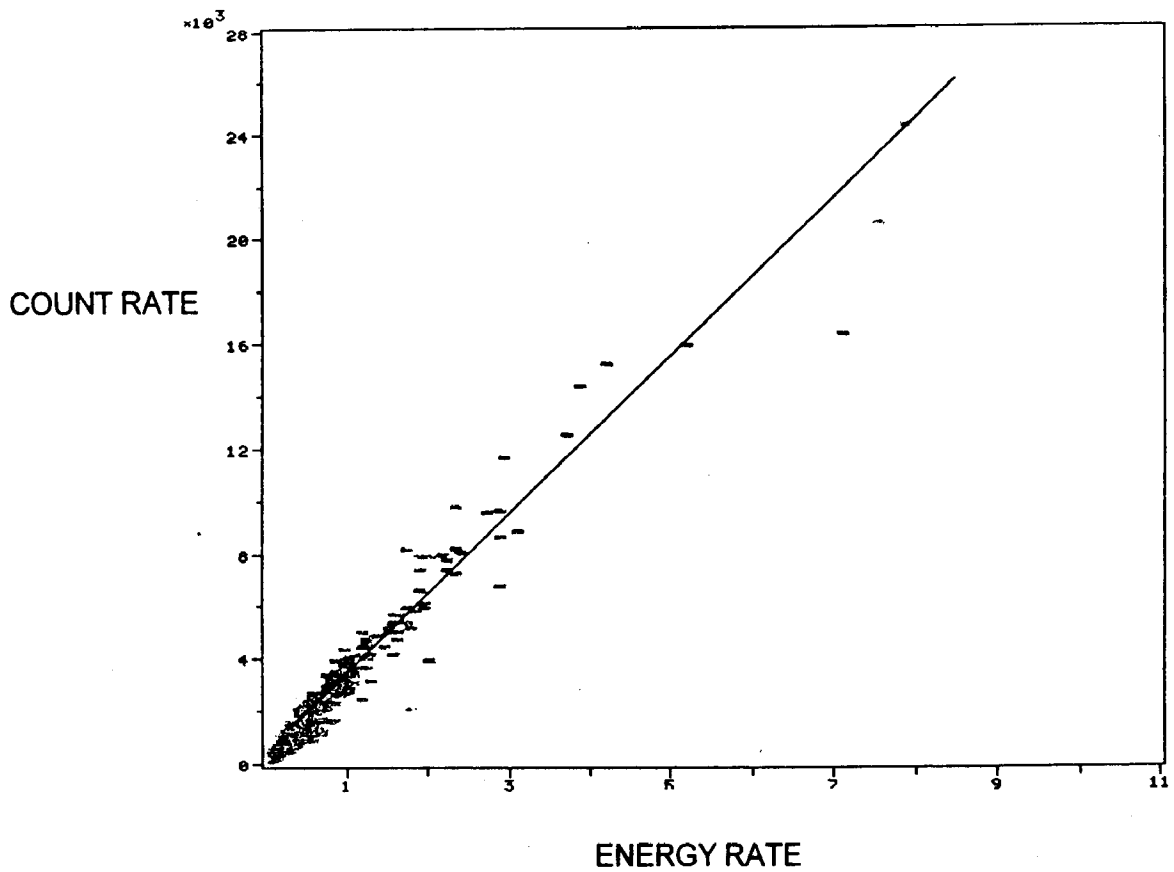


FIGURE 5. - The linear relationship between count rate and energy rate.

a. 3.5 hours

b. 48 hours



FIGURE 6. - The cross-sectional views of leather samples.

shows a more pronounced saw shape pattern for the hit rate profile. The 3.5-hour sample fiber bundles, on the other hand, due to a poorly opened up structure, do not have enough freedom to aggregate together; instead the stress is more evenly increased as the sample is being stretched, resulting in less fluctuation in the hit rate, as shown in Figure 2a.

Acoustic Frequency Patterns

We also examined the acoustic frequency response to the change of liming time. Figure 3 illustrates the change of frequency as the tensile test proceeds. Figure 3a demonstrates that the 3.5-hour liming sample emits a high pitch sound initially; however the frequency decreases progressively up to the end of the tensile test. In contrast, a longer hour liming sample, as shown in Figure 3b, maintains a relatively stable frequency throughout the tensile test. Moreover, for the short liming time samples (Figure 3a), it appears that an earlier fracture has occurred at around 20 seconds as indicated by the steep decrease in frequency. This is probably due to a high amount of residual non-collagenous material remaining in the short time liming sample, which has caused a stress concentration and consequently made the fiber bundles prematurely fracture. This behavior was not observed in the longer liming sample (Figure 3b), which had most of the non-collagenous materials removed, rendering a more flexible structure to the leather. Therefore, the sound frequency remained relatively stable throughout the tensile test.

Acoustic Count Rate

Figure 4 illustrates the AE count rate-history produced during tensile stretch. Counts refer to the number of acoustic oscillations (waves), whereas "hits" mean the number of acoustic pulses. One acoustic pulse can consist of many counts, dependent on the intensity of the wavetrain. The stronger or more energetic the wavetrain, the higher the number of counts. Therefore, the number of counts is a function of the magnitude of acoustic energy. This relationship can be illustrated in Figure 5, which shows a typical linear correlation between the acoustic count rate and the energy rate.

As demonstrated in Figure 4, the difference in count-rate patterns is much more significant than those from hit-rate or frequency comparisons. It clearly illustrates that the sample with a longer liming time shows a more erratic pattern in count-rate, as shown in Figure 4b. The reason behind this behavior was described previously in the section describing the hit-rate pattern observations. This may be elucidated

better by SEM photos. Figure 6a shows the cross-sectional view of the 3.5-hour liming sample, which has a distinctively denser structure than that of the 48-hour liming sample shown in Figure 6b, where the structure shows more space between the fibers. Additional examples are shown in Figure 7, indicating as the liming time increases from 7 hours (Figure 7a) to 24 hours (Figure 7b) and then to 96 hours (Figure 7c), the count-rate saw-shaped patterns become more and more evident.

Cumulative Counts

As illustrated in previous figures, a rate plot of AE counts can highlight the changes in AE activity that occur during the tensile test. Observations so far have shown that the count-rate history patterns correlate well with the degree of opening up of the fibrous structure. However, a quantitative correlation between an acoustic quantity and the degree of opening up would be more desirable. We then investigated the cumulative counts as a function of liming time. The relationship between the cumulative counts and count rate can be written as follows.

$$\sum_0^t N = \int_0^t \mathcal{R} \cdot dt \quad (2)$$

Where:

ΣN = cumulative count;

\mathcal{R} = count rate;

t = time

Figure 8 shows a typical plot of the cumulative AE counts, i.e., the summation of counts measured since the start of the tensile test up to the fracture of the test samples, as a function of time. These curves propagate very similarly to those of stress-strain curves. The cumulative counts slowly increase with stretching time until the leather is fractured. The plot of Figure 8 provides a convenient format for reading off a total emission quantity. The total counts of a leather sample are strongly associated with the total acoustic energy released from its deformation, such as tensile fracture. The more acoustic energy generated in a tensile test, the more acoustic counts are emitted. This relationship is demonstrated in Figure 9, which shows the linear correlation between these two acoustic quantities.

Figure 10 plots the total counts (the cumulative counts read off as sample fractured) as a function of liming hours. The total counts increase drastically as the liming time increases from 3.5-hours, to 7-hours and then to 24 hours. However, there is a sharp drop in total counts as the liming time increases to 48 hours; thereafter a slow increase was

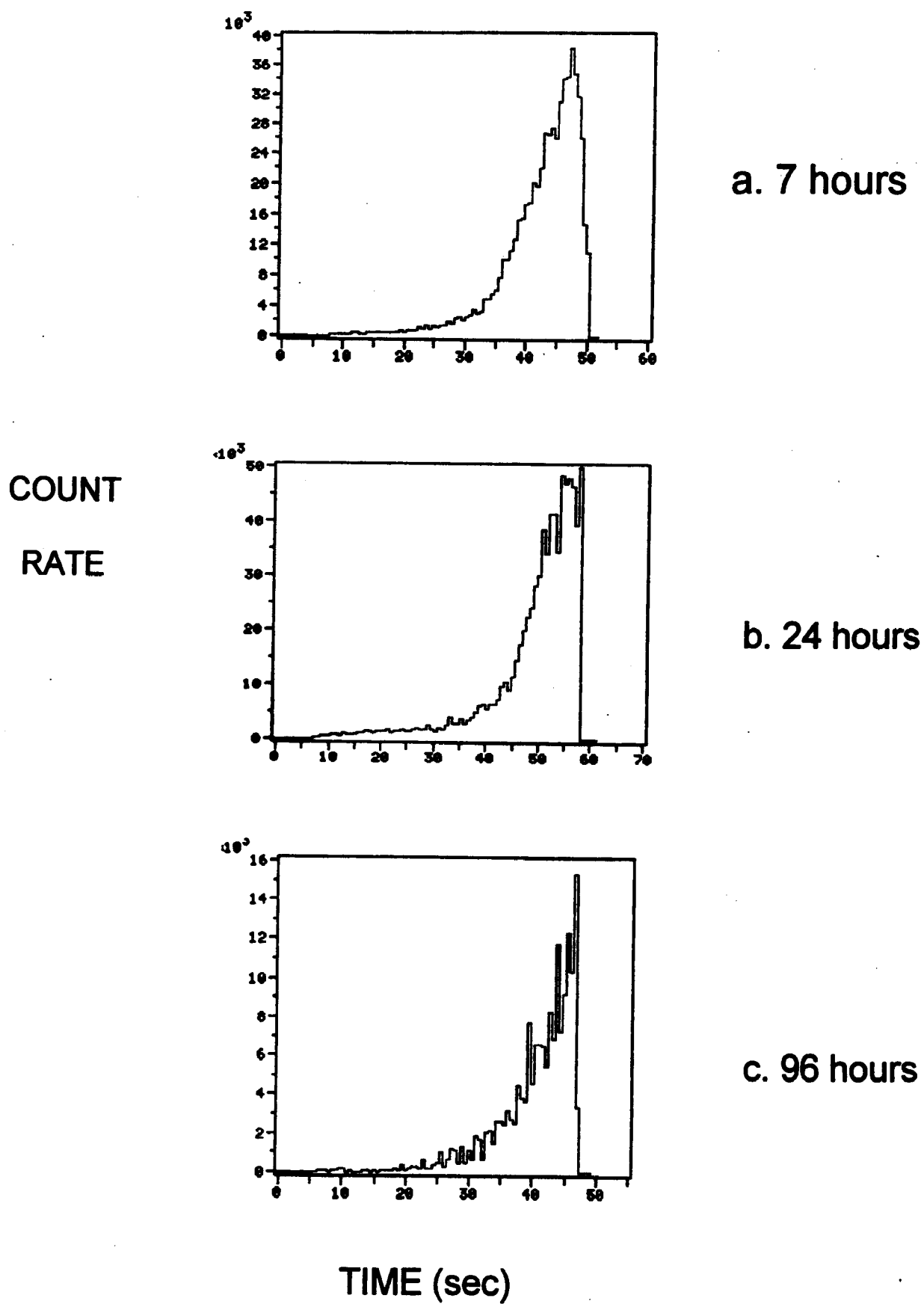


FIGURE 7. - Acoustic counts history.

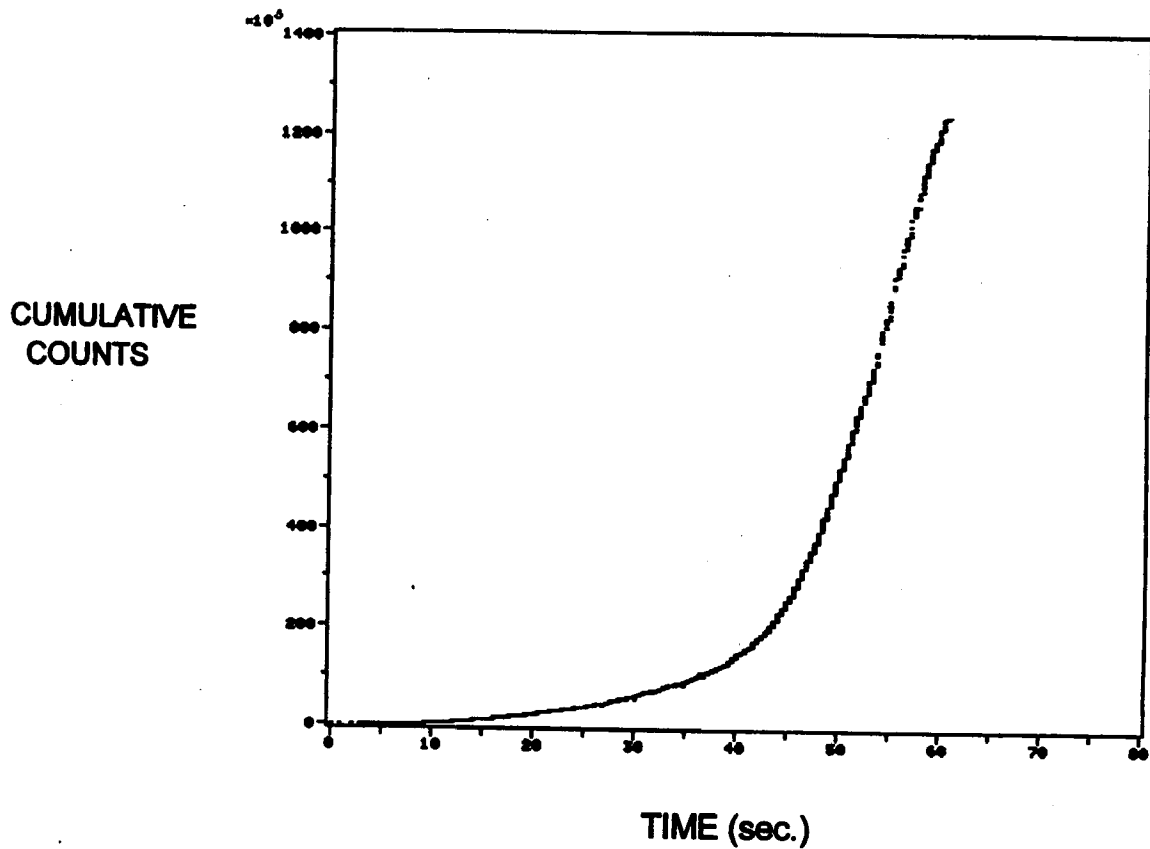


FIGURE 8 - Cumulative plot of acoustic counts to obtain the total acoustic counts.

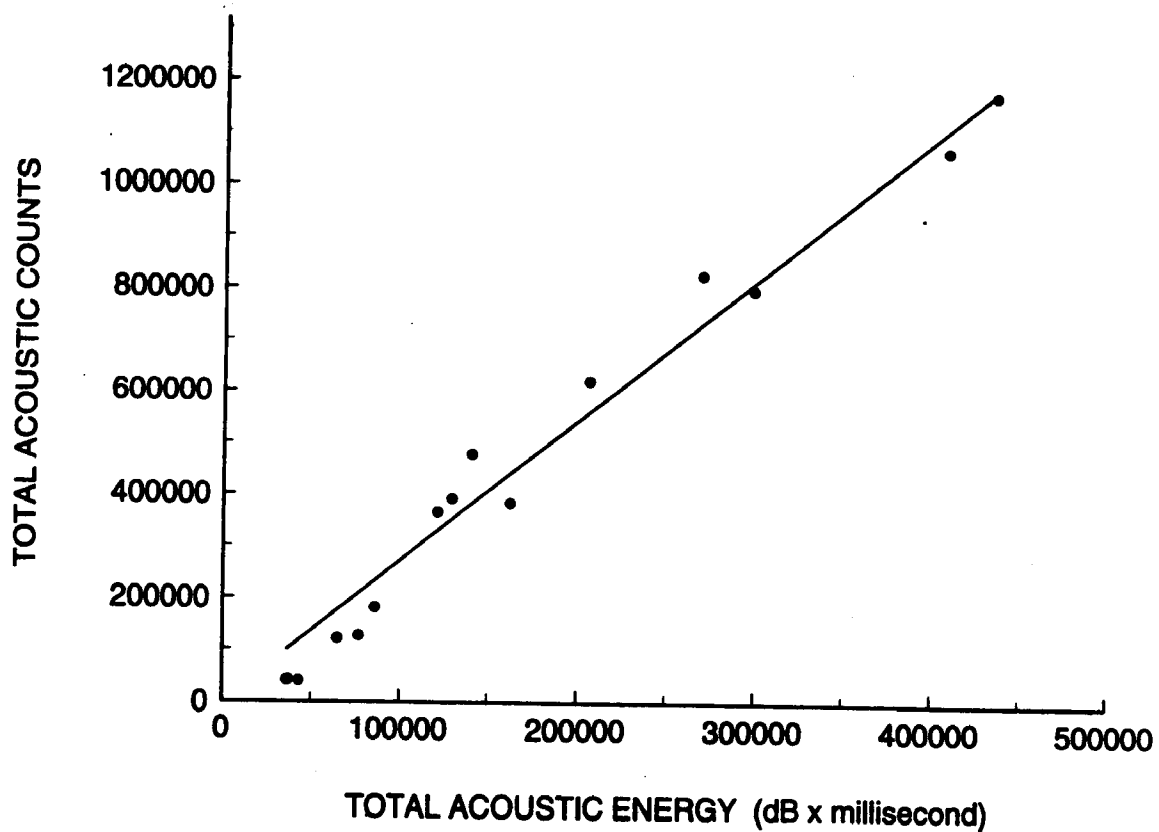


FIGURE 9. - The linear relationship between the total acoustic counts and energy.

observed as the liming time increased to 72 hours and to 96 hours. The reason for the first sharp increase in total counts is ascribable to a significant increase in fiber movements and wave transmissions that in turn emit more sound from the test sample as it is subjected to a tensile stretch. The action of liming as mentioned previously is to open up the fiber structure. The longer the liming, the more effective the removal of non-collagenous components and the higher the extent of fiber bundle splitting in the hide, as liming time increases from 3.5 to 24 hours. However when liming is prolonged for more than 24 hours, the fiber structure opens up to such an extent that it leads to a drastic increase in the penetration of fatliquor during the subsequent fatliquoring process. Lubrication effects of fatliquor can result in a decrease in tensile stress during the tensile test, consequently producing less sound. This argument may elucidate the drastic decrease in total AE counts observed for the 48-hour, 72-hour and 96-hour liming samples. However, a small increase still occurs in AE counts as the liming time increases from 48 to 96 hours in those samples. This may be attributable to the effects of more fiber bundles splitting.

Tensile strength

Tensile strength is one of the most important qualities of

leather. Figure 11 shows the resultant tensile strength as a function of liming time. It demonstrates that the tensile strength of leather increases drastically from 3.5 hours to 7 hours liming; hereafter, the tensile strength changes very little. Presumably, when the hides are limed for a short duration there are still a great amount of impurities imbedded in the leather, such as glue-like non-collagenase materials, and the fibers do not split sufficiently. This results in a hindrance of fiber movements, which leads to stress concentration and consequently a poor tensile strength.

Toughness

Toughness has been described in a previous report as a quantity associated with the energy required to fracture leather.⁸ We have characterized the fracture resistance of leather by measuring the energy needed to fracture a sample, which is the area under the force-elongation curve. Good fracture energy generally reflects a superior balance of strength and flexibility. Our previous investigation demonstrated a strong correlation between tear strength and fracture energy.¹⁰ Figure 12 shows the resultant toughness of leather as a function of liming hours. The toughness drastically increases as the liming time increases from 3.5 hours to 7 hours, and peaks at 17 hours. Then it starts to decrease somewhat, when the liming time is further

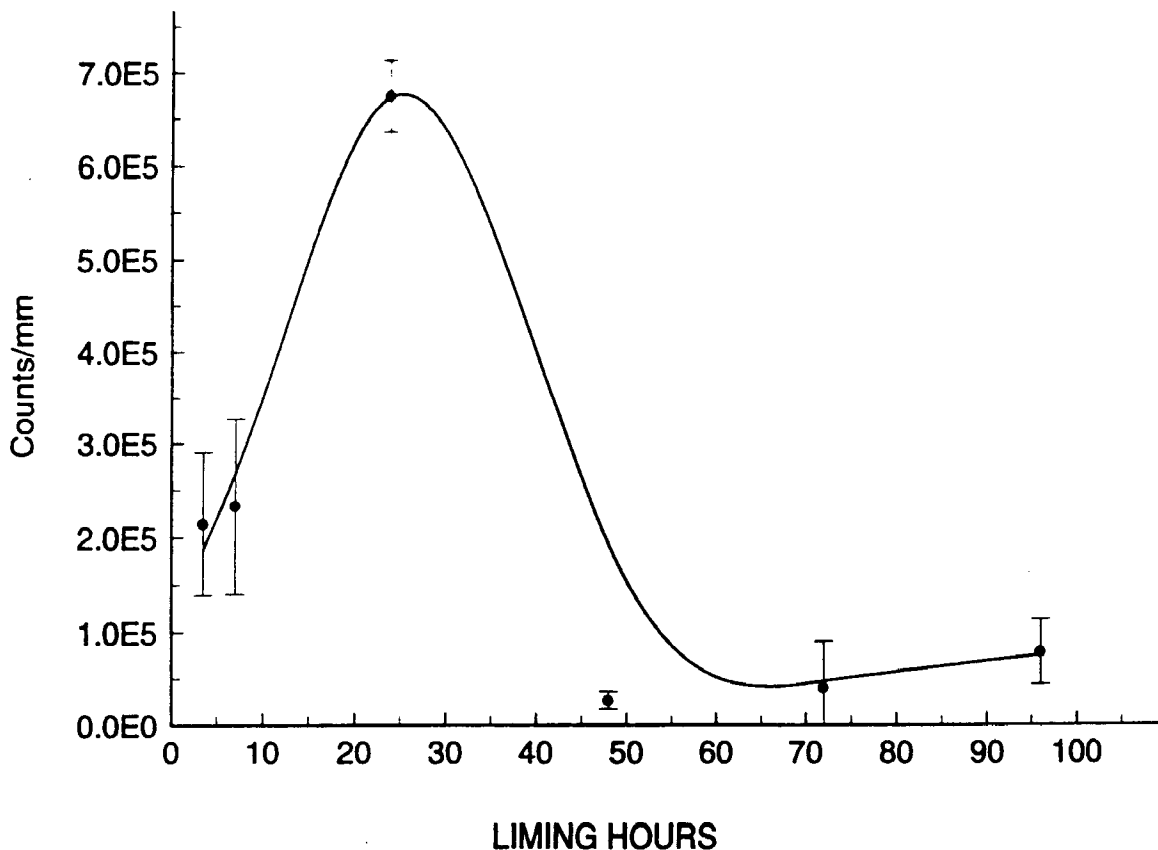


FIGURE 10. - Total acoustic counts vs. liming time.

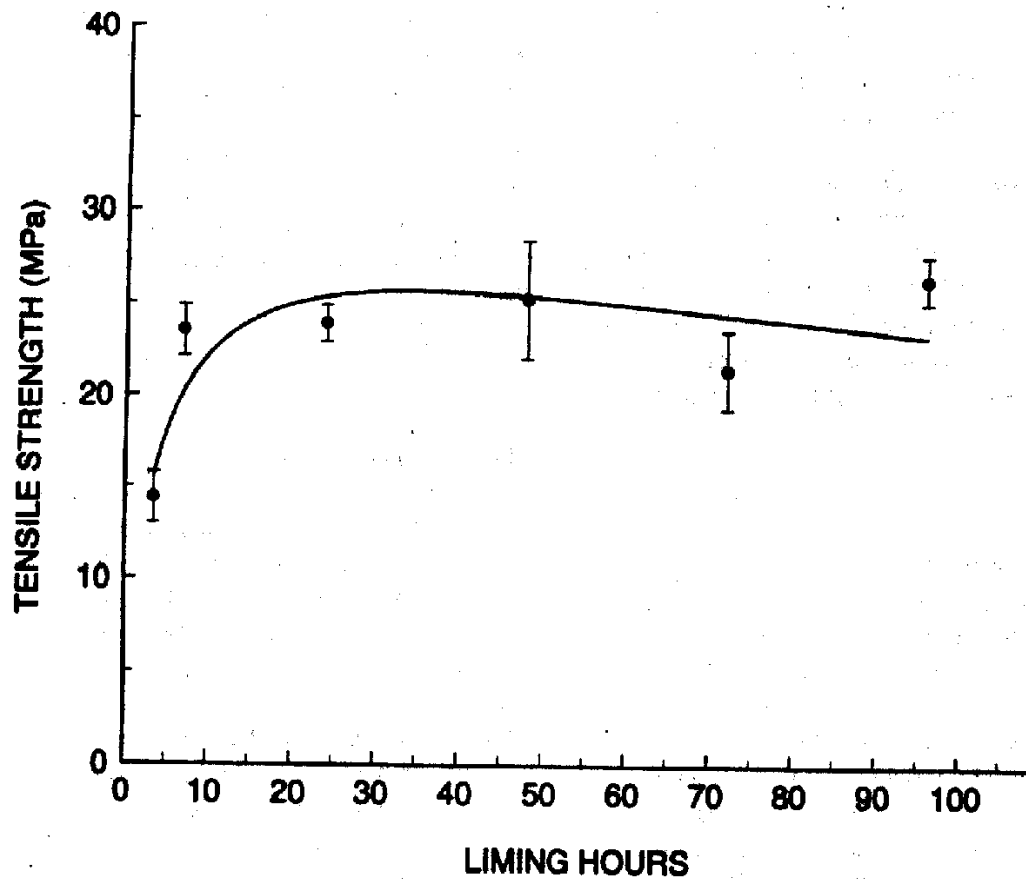


FIGURE 11 - Effects of liming time on the resultant tensile strength.

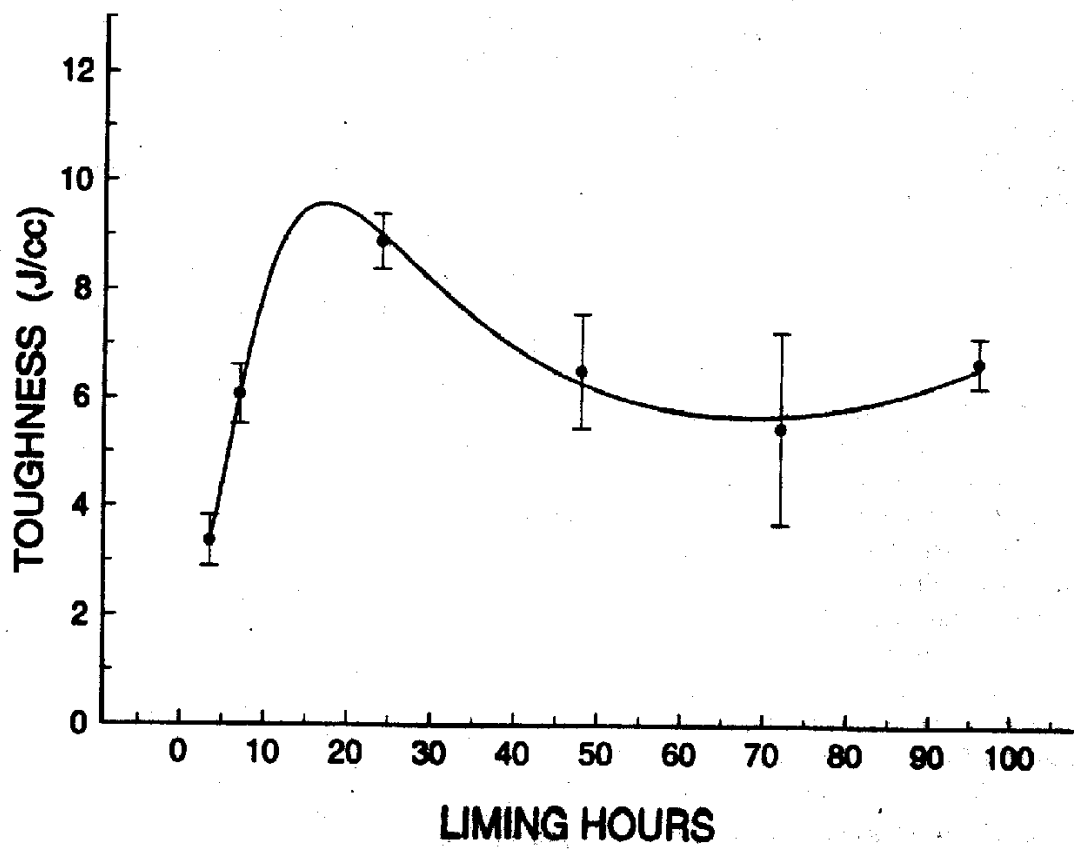


FIGURE 12. - Effects of liming time on the resultant toughness.

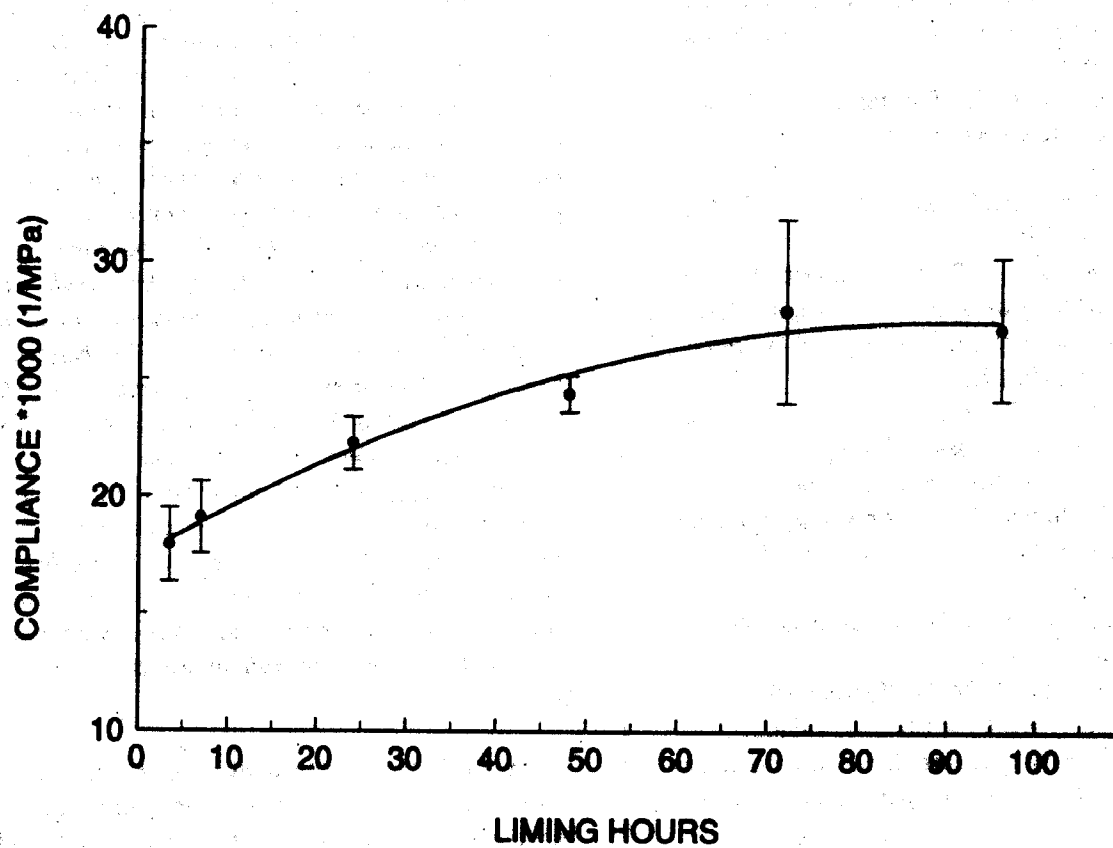


FIGURE 13. - Effects of liming time on the resultant compliance.

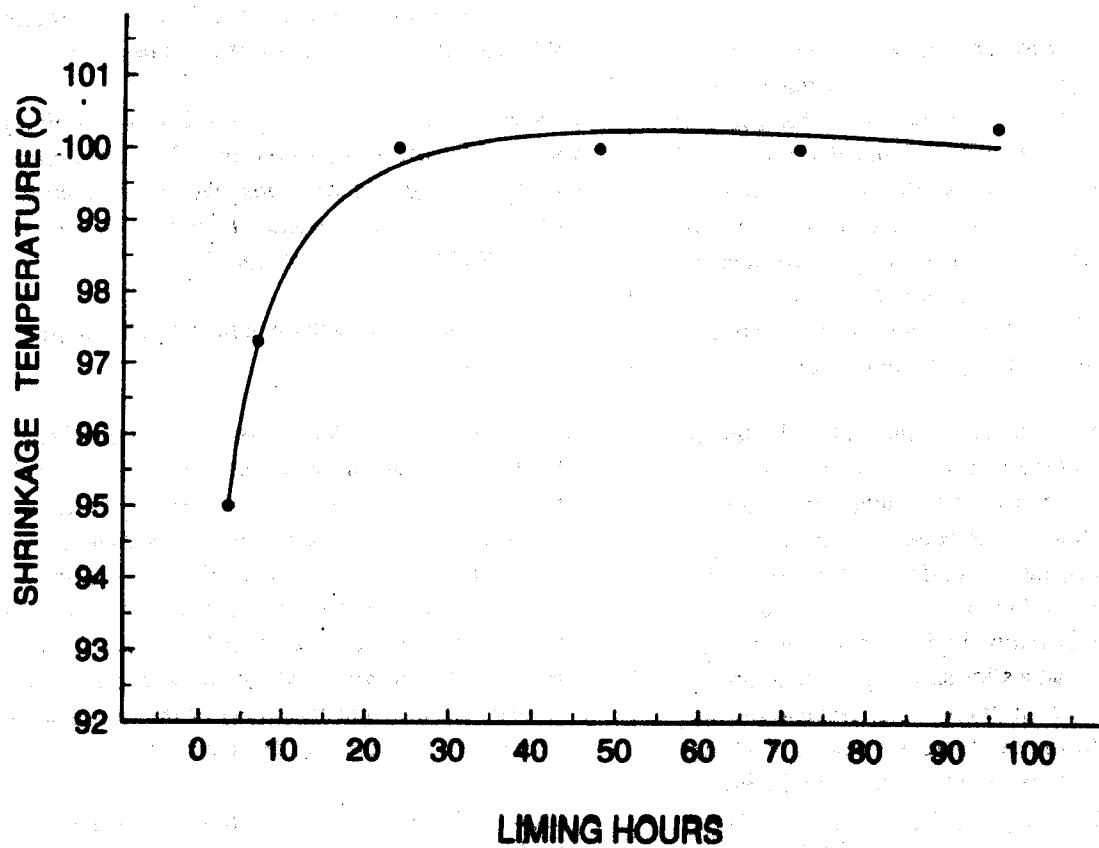


FIGURE 14. - Shrinkage temperature changes with liming time.

increased. The early sharp increase in toughness with liming time can be attributable to the increase of freedom in the fiber bundle movements due to a more effective removal of non-collagenous materials from the leather. This increase of freedom in the fiber bundles enables fiber bundles to slip over each other and makes them more uniformly share the tensile stress in the tensile test, thereby increasing the fracture resistance of leather. However, when the liming action is prolonged past a certain time, fiber splitting may go too far and undermine the integrity of the fibers. Consequently, the fracture resistance of the leather structure may decrease, as shown in Figure 12, when the liming time is increased above 24 hours.

Compliance

Adequate compliance or pliability is a very important quality requirement for certain leather products, particularly for garments, upholstery, and footwear. It provides comfort and a good "handle" to the user. The quantitative assessment of compliance or its reverse term "stiffness" can be based on measurements of the resistance to a small deformation by tensile stress. The resistance may be quantitatively represented best by the initial slope of the load-displacement curves or the stress-strain curves in the elastic deformation region, i.e., the Young's modulus. It is commonly known that the higher the Young's modulus, the less compliant the leather is. In fact, its reciprocal is named "compliance."¹¹ As demonstrated in Figure 13, data show that leather with a longer liming time yields a higher compliance, i.e., softer leather. This result agrees with the action of liming, as mentioned before. The longer the liming, the more opening up or fibers splitting may take place in the leather. Consequently, the friction resistance associated with the fiber movement is greatly reduced, thus improving the compliance of the leather.

Shrinkage Temperature

The cross linking between the chrome tanning agent and the hide protein will result in stabilizing the collagen fibers. The most common method to determine the degree of stabilization is by measuring the shrinkage temperature of leather, as described in the experimental section. The action of lime, besides opening up the fiber structure, disrupts some of the hydrogen bonding between the adjacent protein chains and brings on chemical changes to collagen by the hydrolysis of amide side chains to carboxyl groups. Parallel with this will be an increase in chemical activity of the hide and an effect on the degree of stabilization of collagen fibers by chrome fixation. Figure 14 demonstrates the effect of liming time on the shrinkage temperature of the

resultant leather. The shrinkage temperature increases sharply as the liming time increases from 3.5 hours to 24 hours; thereafter, the liming time no longer produces a significant change in shrinkage temperature. This result implies that prolonged lime action up to 96 hours does not chemically modify the collagen to an extent that would cause degradation or denaturation of the collagen in leather. Otherwise it would show a sharp drop in the shrinkage temperature.

CONCLUSIONS

This investigation has demonstrated the usefulness of the AE method to characterize the degree of opening up of the fibrous structure of leather made with various liming times. We conclude that a qualitative correlation exists between the degree of opening up of leather and the acoustic counts measured by an AE analyzer. The results of this work may provide a route to monitor the degree of opening up of leather, which previously was almost unmeasurable. Observations also show that the prevailing liming time used by most tanneries, i.e., 24 hours, produces an adequate degree of opening up in the leather reflected from the AE measurements along with a high shrinkage temperature of 100 °C. This prevailing liming time also yields the highest toughness. However, if leather products require better compliance, then the liming time may need to be extended past the standard 24 hours in order to achieve a higher degree of opening up in order to reduce the friction of collagen fibers in the leather.

ACKNOWLEDGMENTS

The authors wish to thank Peter H. Cooke and Doug Soroka for the SEM micrographs. Particular appreciation is extended to Paul Kronick, Satyendra De, and David Coffin for their invaluable suggestions, and to Maryann Taylor for her time and help with the shrinkage temperature measurements. We would also like to thank Amanda Long and Dave Langridge from the British Leather Confederation for their help in providing the leather samples for initial evaluation.

REFERENCES

1. Kronick, P. L., and Thayer, P.; Fiber adhesion in solvent-dried calfskin studied by acoustic emission spectroscopy. *JALCA* **84**, 257-265, 1989.
2. Kronick, P. L., and Maleeff, B.; Nondestructive failure testing of bovine leather by acoustic emission. *JALCA* **87**, 259-265, 1992.

3. Liu, C.-K., and McClintick, M. D.; The use of acoustic emission in predicting the tensile strength of leather. *JALCA* 94, 8-19, 1999.
4. Liu, C.-K., and DiMaio, G. L.; Tear resistance of leather characterized by acoustic emission. *JALCA* 95, 170-178, 2000.
5. Pollock, A. A.; Metals Hand Book, 9th Ed. V.17, ASM International, 278-294, 1989.
6. Harris, D. O., Tetelman, A. S., and Darwish, F. A. I.; Detection of fiber cracking by acoustic emission. ASTM STP 505; American Society for Testing and Materials, Philadelphia, P.A., 238-249, 1972.
7. Miller, R. K., McIntire, P.; eds., Nondestructive testing handbook, Vol. 5: Acoustic Emission Testing, 2nd edition, American Society for Nondestructive Testing, pp. 29, 1987.
8. Liu, C.-K., and McClintick, M. D.; An energy approach to the characterization of the fracture resistance of leather. *JALCA* 92, 103-118, 1997.
9. Fein, M.L.; Determination of Ts on suspended leather specimens. *JALCA* 60, 15-30, 1965
10. Liu, C.-K., and McClintick, M. D.; Tearing behavior of chrome-tanned leather. *JALCA* 94, 129-145, 1999.
11. Mase, G. E.; Theory and problems of continuum mechanics, McGraw-Hill Book Company, New York, pp. 201, 1970.

CONVENTION DISCUSSION

Dennis Shelly, Leather Research Institute It occurs to me that the extension or stress on the leather and the resulting acoustic signal comes from a combination of fiber slip and perhaps fiber breakage. It seems to me that those two categories of acoustic signals should be able to be differentiated in your signal with this liming study and the degree of openness that you create in the hide. Could you comment on to what extent you think the liming operation and the opening up would have on the relative amounts of fiber slip and fiber breakage during the stressing of the leather?

Fiber breakage is occurring mostly at the end of the test so you can see a big peak at the end of the test. You see a big jump in the test results (i.e. acoustic counts). But slippage of the fibers- fiber friction all the noise coming from there happens in the beginning and the middle of the test but not at the final stage of the test. On the other hand, the acoustic energy from breakage is really high energy and we can detect that. I think that I had a slide showing the relationship between the count rate and the energy rate. You can see that there is a linear line. At the top there are several dots that actually belong to the fiber breaking or fiber bundle breakage. You raised a very good point. I think that we should take a closer look at that - a very good research direction.

Steve Wren, Shill & Sheilacher/Struktol: I was interested to see your use of the shrinkage temperature. That was obviously on the tanned and finished leather. I have used shrinkage temperature in liming work as a control in the beam house where you take lime pelt and do a shrinkage temperature directly on the lime pelt. There was at one time an official method suggested by a guy called Gray Brooks going back to the 50's or 60's where the shrinkage temperature was established by deliming the sample with buffer and then doing the shrinkage. You can also take the limed pelt and do a shrinkage temperature. The better the liming the lower will be the shrinkage temperature gets. This varies between the high 60's down to the high 40's. This method although a fairly simple control method, is perhaps not very accurate. I suggest that it might be interesting exercise to compare those shrinkage temperatures with the work that you are doing which seems to be more controlled and accurate. I think that there is need for a test in liming that the tanner can run on the same day or the day after the process is completed. We have to wait six weeks for a sample in the liming.

Your point is correct. My study is on the shrinkage temperature of leather. It is not trying to see stabilization of the collagen. I was trying to see whether there was degradation in the leather because I was worried about using 96 hours or 4 days for liming. What I see is that you get very good properties even after 96 hours of liming. The problem that I focused on is whether your leather is degraded.

T. Ramasami, CLRI: During the acoustic emission study, you have looked at the emission. What is the percentage of the acoustic signal that is absorbed by your matrix? When you give an acoustic signal, leather is a very lossy substance. It absorbs acoustic signals.

This is related to the sensor area. You are talking about the sensor area?

T. Ramasami, CLRI: No. When you give an ultrasound signal, there is absorption, there is scattering, and there is emission. You have been able to monitor the acoustic emissions through a signal detection system. Leather happens to absorb a significant proportion of acoustic signals and creates the kind of hyper-slippages that one of my fellow colleagues was pointing out earlier. If the energy you have supplied is predominantly used for the absorption and slippage phenomenon, then the sensitivity of the technique would be hampered by the nature of the substrate.

I know what you mean. The sample that we are testing is 2 mm thick and we use a very, very sensitive sensor. So the

damping factor is very low.

T. Ramasami, CLRI: Does creep give you any problem with this measurement? Basically leather has a composite character. It will have two fiber axes wound around each other and they will open up. Is there any creep phenomenon in your study at all?

(Response to this question was not given by the author at the convention) There is no creep phenomenon in our study because the samples were subjected to a constant strain rate, not a constant stress. One thing that I would like to clarify is that we are not sending acoustic signals into the leather samples. Instead we are measuring acoustic signals generated by stretching the leather in the tensile test.
